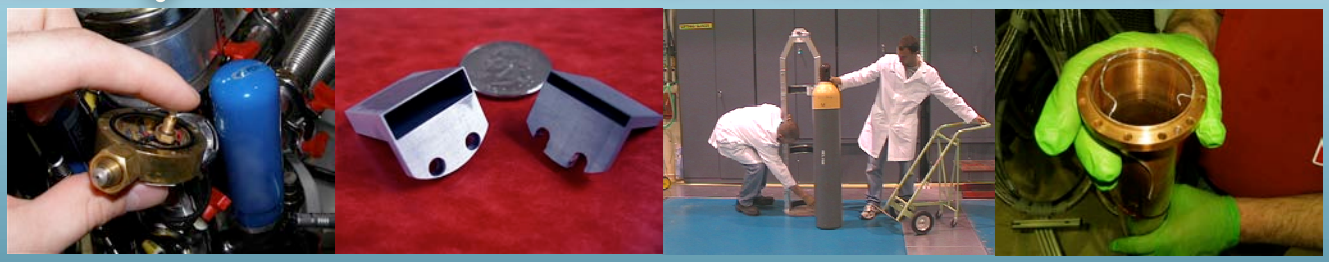
Sample Environment News



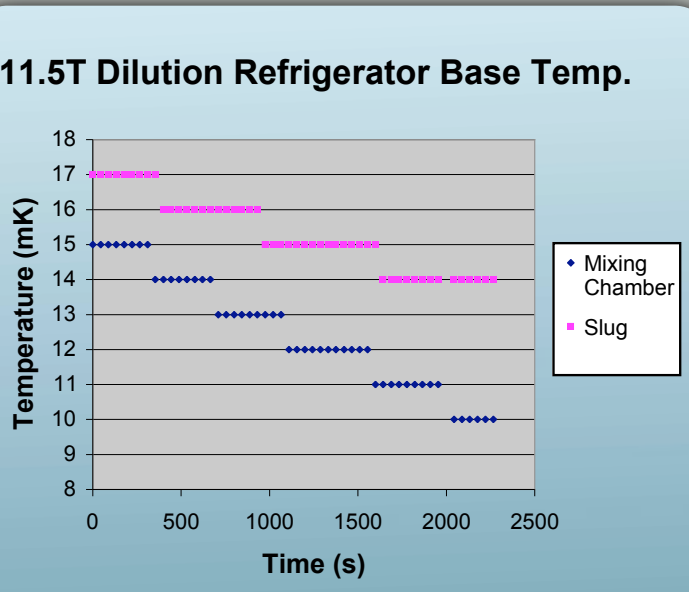
http://www.ncnr.nist.gov/equipment/ancequip.html

June 6, 2007

Dilution Refrigerator Reaches Record Low

Over the past few years the base temperature for the dilution refrigerator in the 11.5T Oxford magnet has gone nowhere but up.* At the same time the amount of ^{3}He/^{4}He mixture that runs through it has gone nowhere but down. We expected that by adding mixture, in some undetermined proportion, we could decrease the base temperature, thereby reversing both trends.

First we needed to know what we had in the mixture. For the chosen method, residual gas analysis (RGA), Tom Gentile generously provided the setup and the expertise on its use. After comparing the amounts of ^{3}He and ^{4}He that we currently had with the known original amounts, we knew how much needed to be added. Evan Fitzgerald added the gases separately and then the mixture was cleaned by running it through a liquid nitrogen cold trap before the system was cooled for its big test.



The base temperature had been anywhere from 45 – 60mK before adjusting the mixture. In the original commissioning of the system it had been 18mK. After our adjustment of the mixture, the base temperature as measured on the slug sensor (nearest the sample) was 14mK and on the mixing chamber (the sensor closest to the source of cooling) reached 10mK. After eight years of service, the dilution fridge is running better than when it first arrived!

Gas Cylinder Movie Series Released

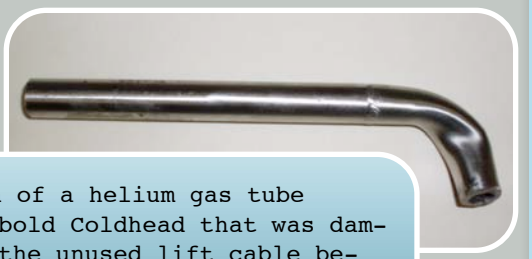
Sarah McKenney has produced two additional "how-to" movies for your viewing pleasure: *Working with Gas Cylinders* and *Transporting Gas Cylinders*. These action-packed movies guide their audience through most encounters with gas cylinders. *Working with Gas Cylinders* highlights the critical aspects of gas regulator operation, depicts safe handling of cylinders, and shows how to move a regulator from an empty to a full cylinder. *Transporting Gas Cylinders* identifies gas cylinder storage areas, explains how to transport and lift cylinders, and discusses methods of securing cylinders. It also describes the protocol for obtaining and working with specialty and flammable gases. Visit the sample environment website's "How-to Videos" subsection to learn about gas cylinder safety in the video format of your choice: http://www.ncnr.nist.gov/equipment/ancequip.html

*Certain commercial equipment is identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment identified is necessarily the best available for the purpose.

Need a Lift?

Bill Clow has made it easier to safely crane lift sample environment equipment. You will find color-coded, pre-rigged lifting assemblies supplied at instruments with individual cranes and in C-100's sling cabinet. The same color coding has been applied to the lifting eyes on Sample Environment carts and equipment. This color-coding designates one, two, three, and four-point lifting assemblies. This was done to prevent an unused lift cable from becoming tangled in the wiring, tubing and valves that are on top of our equipment. An entangled cable can cause the lift to become unbalanced, plus it can bend or pull off parts of the equipment. For example, avoid using a four-point lifting assembly on a piece of sample environment equipment with three lifting points.

There are still a few pieces of equipment that are not color-coded and require basket lifts by a skilled technician. Please contact George Baltic's or Sample Environment's staff for such a lift or if you ever have any concerns about moving an object with a crane.



A section of a helium gas tube from a Leybold Coldhead that was damaged when the unused lift cable became entangled while lifting. Notice the kinked bend on the right end.

Where Should I Put My Sample?

Sample cans, sample cans. Has anyone seen any sample cans? We have made a concerted effort to build up our stock of commonly used sample cans.

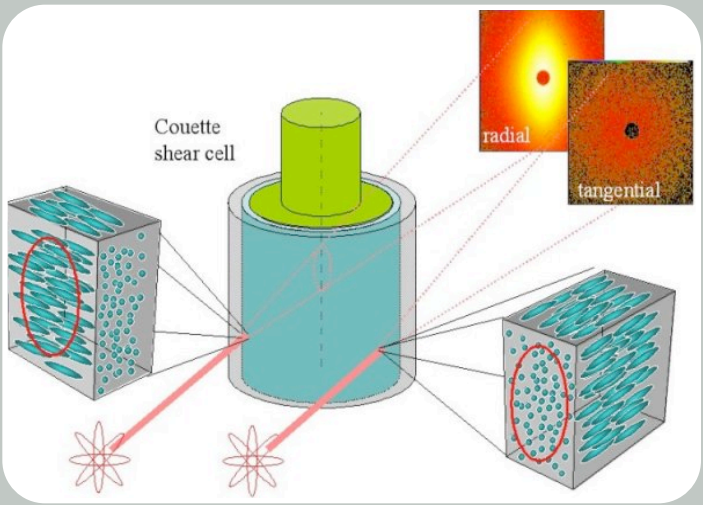
In particular, the BT-1 vanadium powder sample cans have increased their stores 2.5-3 times over what we had previously. For each of the sizes A through E (sample volumes 1.5 – 11.5 cc), we now have 40-50 sample cans that can be used. In addition, there are now three lid styles to choose from. These lids allow attaching to sample environment equipment through a stud on the lid (room temperature sample changer), 4 bolt attachment (bottom-loading CCRs), or a threaded socket on the lid (orange cryostats, top-loading CCRs).

For the other instruments, we have additional aluminum powder sample cans, small and large single crystal sample cans, and for the first time here at NIST we have made a substantial quantity of the Brookhaven-style single crystal sample can. Thanks to a contracting effort by our engineering group, we can replenish low supplies on a time scale of two months for any design.

The New SANS Rheometer

The SANS group has developed a device, named the "RHEO-SANS", capable of probing the shear rheology while simultaneously probing the material micro structure through SANS. This new device is expected to provide valuable insight into shear-induced structure in a broad class of deformable systems, including polymers, liquid crystals, surfactant mesophases, nanocomposites, emulsions and colloidal systems.

The RHEO-SANS device consists of a Couette-type flow cell retrofitted to a commercially available rheometer. A Couette flow cell features two concentric cylinders, one in rotation and the other stationary. The RHEO-SANS device fixes the outer cylinder, referred to as the cup, and rotates the inner cylinder, often called the bob. The cup and bob are constructed of either quartz or titanium for neutron transparency.

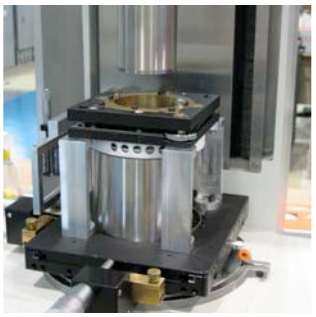


The Couette cell geometry allows for neutron scattering experiments to be performed with the incident neutron beam parallel to the velocity gradient, called the radial configuration, or parallel to the flow direction, called the tangential configuration. Structural information can then be garnered parallel and perpendicular to the direction of the fluid flow, for complete microstructural characterization.

Many types of rheological tests can be performed while simultaneously recording neutron scattering data. Simple shear flow can be carried out at either a constant shear stress or constant shear rate. Also, creep shear as well as creep recovery are available. The newest feature is the ability to perform amplitude oscillatory shear deformation. Time dependent fields, such as oscillatory and flow start-up, can be generated and studied, but advanced neutron scattering techniques are required to overcome neutron flux limitations.

Couette cell gap sizes of 0.5 and 1.0 mm are currently available requiring sample volumes of 8 and 12 ml, respectively. A small volume cell (<4ml) is under development. The sample temperature can be controlled from -40 to 150ºC and a solvent trap is available to prevent sample evaporation. The technical specs in the table above provide a breakdown of the shear capabilities.

RHEO-SANS
CHECK IT OUT



Accuracy	1% of maximum value
Maximum Torque	150 mNm
Torque Resolution	0.01 μNm
Speed Range	10^{-4} to 1000 min^{-1}
Shear Rate Range	$1.3*10^{-4}$ to $4.8*10^{3}$ s^{-1}
Shear Stress Range	0.001 to 512 Pa
Viscosity Range	$1.7*10^{-3}$ to $2.7*10^{8}$ Pas
Temperature Range	-40 to 150 °C

For more information or if you wish to use the rheometer, please contact:

Lionel Porcar
porcarl@nist.gov, x5049
Jeff Krzywon
jkrzywon@nist.gov, x6650

Up to 10.0 kbar Pressure Cells Available

After a two year long development process we have extended the NCNR's hydrostatic pressure capabilities from a maximum of 4.2 kbar to 10 kbar. This process included a detailed material analysis of candidate metals, encompassing neutron properties as well as possible activation. Assessing the viability of manufacture and the intricacies of the design specifications was accomplished through countless meetings with the vendor. The results of this effort are the three 6 kbar and three 10 kbar pressure vessels now available for use. These vessels were designed to work over a wide temperature range and to have a sample cross sectional area commensurate with the NCNR neutron beam.

Maximum Pressure	6.0 kbar	10.0 kbar
Temperature Range	1.5K to 300K	
Effective Sample Volume	1.5 cm3 (.635 cm dia. x 5.08 cm height of effective illumination)	
Pressurizing Medium	Nitrogen, argon, or helium	
Compatible Equipment	TLCCR, ILL Cryostat	
Total Sample Volume	1.5 cm3	2.2 cm3
Vessel Material	Al 7075-T6	13-8Mo S.S.
Average Neutron Transmission at 2Å	69%	25%
Average Neutron Transmission at 5Å	65%	15%

All of these vessels require a high pressure intensifier for their use. Although there are many vessels now available there is still only one intensifier. Plans are underway for the acquisition of a second intensifier, but in the meantime be certain to plan ahead as experiments are scheduled on a first-come first-served basis. Finally, two important notes when planning and scheduling a high pressure experiment. One, for safety reasons our high pressure intensifier may only be operated by Juscelino Leão. Two, the use of any other pressure cell is forbidden without prior approval from the Sample Environment Team and the NCNR Safety Review Board. Allow at least one month before an experiment for proposal and safety review for experiments using a pressure cell other than our own.

Next Generation Vacuum Pumps

After a review of the "state of the art" in vacuum pumping, we have begun a full-scale migration of all our pumping needs over to a newer "dry" technology. A dry pump completely separates the pump motor oil from the vacuum-pumping chamber. These pumps are being implemented throughout the facility, replacing rotary vane oil pumps. They are also the backing pump on recently acquired turbo pumping stations, thus making these total dry pumping systems.

The new dry technology has a number of advantages. The pump oil cannot become contaminated with water vapor, chemicals, or radioactive contaminants from the act of pumping. Additionally, pump oil can not spit out of a dry pump's exhaust; this is a particularly noticeable difference when initially pumping down from atmospheric pressure. Furthermore, oil never back-streams out of the dry pumps when pumping on low temperature cryostats.

Our current rotary pumps require an oil change four times a year while the new dry pumps require seal replacements once a year. Not only does the general maintenance save time and money for supplies, but repairs are also faster. Rebuilding a rotary pump takes at least two days, but even a failed dry pump can be serviced in an hour.

The dry pump is a scroll type design and quite compact. The capabilities of these pumps range from 6.7 to 35 cubic meters per hour. They are suitable for use on vapor handling processes as well as general vacuum needs. Feedback would be greatly appreciated on their use, reliability, or any additional needs. Please contact Dave Clem for further information.